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BOSTON UNIVERSITY

GRADUATE SCHOOL

A Thesis

THE PREPARATION OF CERTAIN INORGANIC COMPOUNDS

IN A NEW TYPE OF GAS FURNACE

by

Almon Watson Morang

(B.Ch.E., Northeastern University, 1930)

submitted in partial fulfilment of the  
requirements for the degree of

Master of Arts

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## PREFACE

The writer, in preparing this thesis, has freely consulted the literature on the preparation of inorganic compounds by the fusion method in the various types of furnaces, and wishes to express his indebtedness to the writers of this material.

I wish to express my indebtedness to Dr. L. C. Newell for his patience and kindness in directing the laboratory work of this thesis.

1890-1891

1891

1892

The first of the year was a very dry one, and the crops were much injured. The second of the year was a very wet one, and the crops were much injured. The third of the year was a very dry one, and the crops were much injured. The fourth of the year was a very wet one, and the crops were much injured. The fifth of the year was a very dry one, and the crops were much injured. The sixth of the year was a very wet one, and the crops were much injured. The seventh of the year was a very dry one, and the crops were much injured. The eighth of the year was a very wet one, and the crops were much injured. The ninth of the year was a very dry one, and the crops were much injured. The tenth of the year was a very wet one, and the crops were much injured.



TABLE OF CONTENTS

	Page
Preface.....	1
Table of Contents.....	2
Introduction: Methods of Preparing Inorganic Compounds	3
Construction and Operation of the New Furnace.....	7
General Procedure followed in Preparing Compounds in the New Type of Furnace.....	12
Preparation of Calcium Oxalate from Calcium Sulphate..	13
Preparation of Cadmium Sulphide from Cadmium Sulphate.	15
Preparation of Barium Nitrate from Baryte.....	19
Preparation of Barium Hydroxide from Baryte.....	24
Preparation of Strontium Nitrate from Celestite.....	27
Preparation of Strontium Hydroxide from Celestite.....	29
Preparation of Beryllium Oxide from Beryl.....	32
Conclusions.....	36
Bibliography.....	37

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# INTRODUCTION

## METHODS OF PREPARING INORGANIC COMPOUNDS

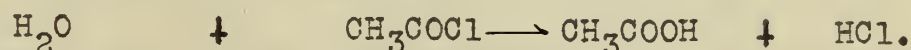
All matter exists in one of three physical phases or in a mixture of these three phases; that is to say, matter exists as a gas, a liquid or a solid or a mixture of these phases.

Chemical reactions are, therefore, limited to these three phases.

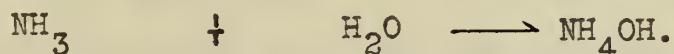
Reactions in the gaseous state are quite simple and may be illustrated by the following reaction:



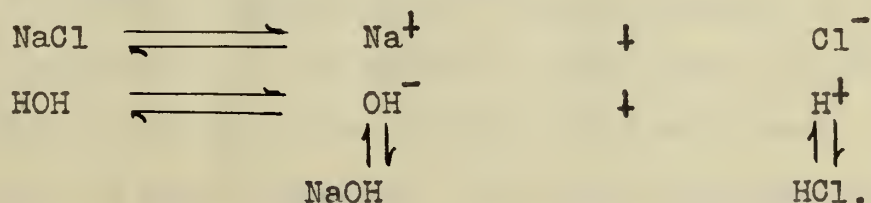
An example of the reaction of one liquid on another:



The reaction of a gas on a liquid:



Of course these reactions are elementary but they serve to illustrate. The reactions of solids dissolved in water and other liquids are quite common. Water is the best known solvent in inorganic chemistry and is very useful in that most inorganic acids, bases, and salts become very highly ionized in a water solution. The following equations represent a simple ionization scheme:



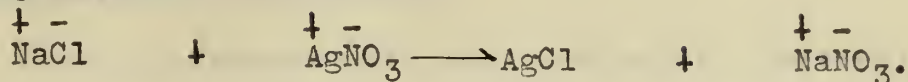
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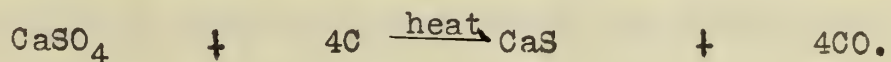
The following equation represents a simple ionization reaction resulting in the formation of a slightly ionized product:



Until now we have concerned ourselves with simple reactions that require no outside help for reaction purposes. It is well known that in order to increase the speed of a chemical reaction one may utilize the following aids: heat, increase contact surface, and introduce a catalyst. In the preceding cases it is not of vital necessity to resort to such means but in the case of two solids reacting it is almost always necessary to increase the contact surface as much as possible and to apply heat. In some reactions of solids the heat is supplied by the exothermic action of the reacting materials. Such reactions have only to be heated to the reacting temperature and they then supply their own heat of reaction until the reacting materials have all been utilized, for example the Thermite reaction:



However, for the most part, solid or fusion reactions require constant heat and a high temperature in order that they may react:



In order to generate the required amount of heat in such a reaction it is necessary to have some sort of a furnace and a great many such devices have been made. All of these devices work quite well, the chief difference in

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them is the method of supplying the heat. The furnace itself is constructed in such a manner as to concentrate the heat on the reacting substances.

Electric furnaces are employed quite frequently and are of three general types: the carbon arc, the electric coil, and the inductance furnace. The carbon arc furnace uses the heat produced between two poles of opposite charge, the electric coil produces heat by the resistance offered to the flow of electricity through a wire, and the inductance furnace supplies heat by the resistance offered to an electrically induced field. These furnaces are all expensive. They are all costly to install and, in the event of a breakdown, expert attention is required.

The type of furnace used in most chemical laboratories is the gas blast. These furnaces are called Fletcher furnaces and most gas blast furnaces are constructed on the same principle as the Fletcher furnace. This type of furnace is simple in its construction and is easily operated. It is made of composition fire brick and bound by a steel shell. The heat is supplied to the furnace by a gas lamp blasted with compressed air. The heat is concentrated on the materials, which are generally contained in a clay crucible which in turn is placed within the furnace on the center of the base, by introducing the flame from the blast lamp into a hole at the base of the furnace. The furnace is cylinder shaped and the top is covered with a disk of fire brick with a small hole in the center. The bottom of the





furnace is also made of fire brick and is permanently attached to the cylinder portion of the furnace, whereas the top is removable so as to allow a method of introducing and removing the clay crucible containing the reacting materials. The flame from the blast lamp hits the sides of the furnace and is reflected in a spiral around the sides of the crucible. Some of the heat is reflected from the top of the furnace, so, but little heat is lost and as a consequence the heat is concentrated on the reacting substances in the clay crucible. This oven is very economical but it requires compressed air and, for small laboratories, this is a disadvantage. It is with this disadvantage, as well as the somewhat lesser disadvantage of the cost of buying the fire brick oven, that a modification of the Fletcher furnace has been made in the form of the new gas oven to be described in this thesis. The modification is applied to both the construction of the furnace and the method of supplying the heat. The experiments herein described are added to prove the worth of the modification both from an economical and chemical viewpoint.



## CONSTRUCTION AND OPERATION OF THE NEW FURNACE

The new furnace consists of four pieces of asbestos paper one eighth of an inch thick as illustrated on page 9. The main part of the furnace is labeled "A". This piece of the furnace is twelve inches on a side with the exception of one corner, which has a square one and a half inches on a side removed in order that a flame may be introduced through it into the cylinder, which is formed by folding the twelve inch square so that the part "X" meets part "X'" and part "Y" meets part "Y'". The seam formed by the meeting edges is sealed by fixing the asbestos strip "B" along the edge from XX' to YY' with water glass. "B" is a strip of asbestos paper ten and a half inches long and one and a half inches wide. Part "C" serves as a cover for the furnace and is a six inch square with a small flap in the center to serve as an outlet. This flap is made by cutting three sides of a one inch square in the center of the six inch square and creasing the fourth side. The last part of the furnace is labeled "D" in the drawing on page 10 and serves as the base for the cylinder.

To assemble the furnace note the drawing on page 10. Part "A", the cylinder portion, sets upright on the base "D", with the edge YY' at the bottom. The part labeled "C" is placed on the top of the cylinder, which is in an upright position. This is the operating position with the square hole formed in the corner of the large square at the base of the furnace. If a piece of copper wire is wound around the





cylinder it will serve to strengthen the furnace as well as maintain the cylinder shape by keeping the seamed edge together.

For best operating conditions, it is well to place the furnace on top of a tripod as illustrated on page 11 supporting it by means of a ring and ring stand as shown in the drawing. The Fischer burner is held in the horizontal position by means of a clamp attached to a ring stand. The flame is introduced into the furnace through the square hole at the base of the cylinder. It is well to use the force of two gas jets in order to obtain as strong a flame as possible. This may be accomplished by attaching the two gas jets to two sides of a "Y" tube and attaching the third side to the burner. The burner should be adjusted mechanically in a vertical position in order to obtain the hottest flame possible and then placed in the horizontal position at such a distance from the crucible that the inner cone of the flame is about one quarter of an inch from the clay crucible within the furnace. This position may be altered at the judgment of the operator to obtain better heating conditions.

The type of crucible generally used is the clay or Hessian crucible. In order that the heat may reach the inner portions of the materials being heated it is well to have a thin walled crucible. The size of the crucible is, of course, limited by the size of the cylinder. The best results were obtained, however, in a Hessian crucible three inches high and two inches wide. In the directions for each of the





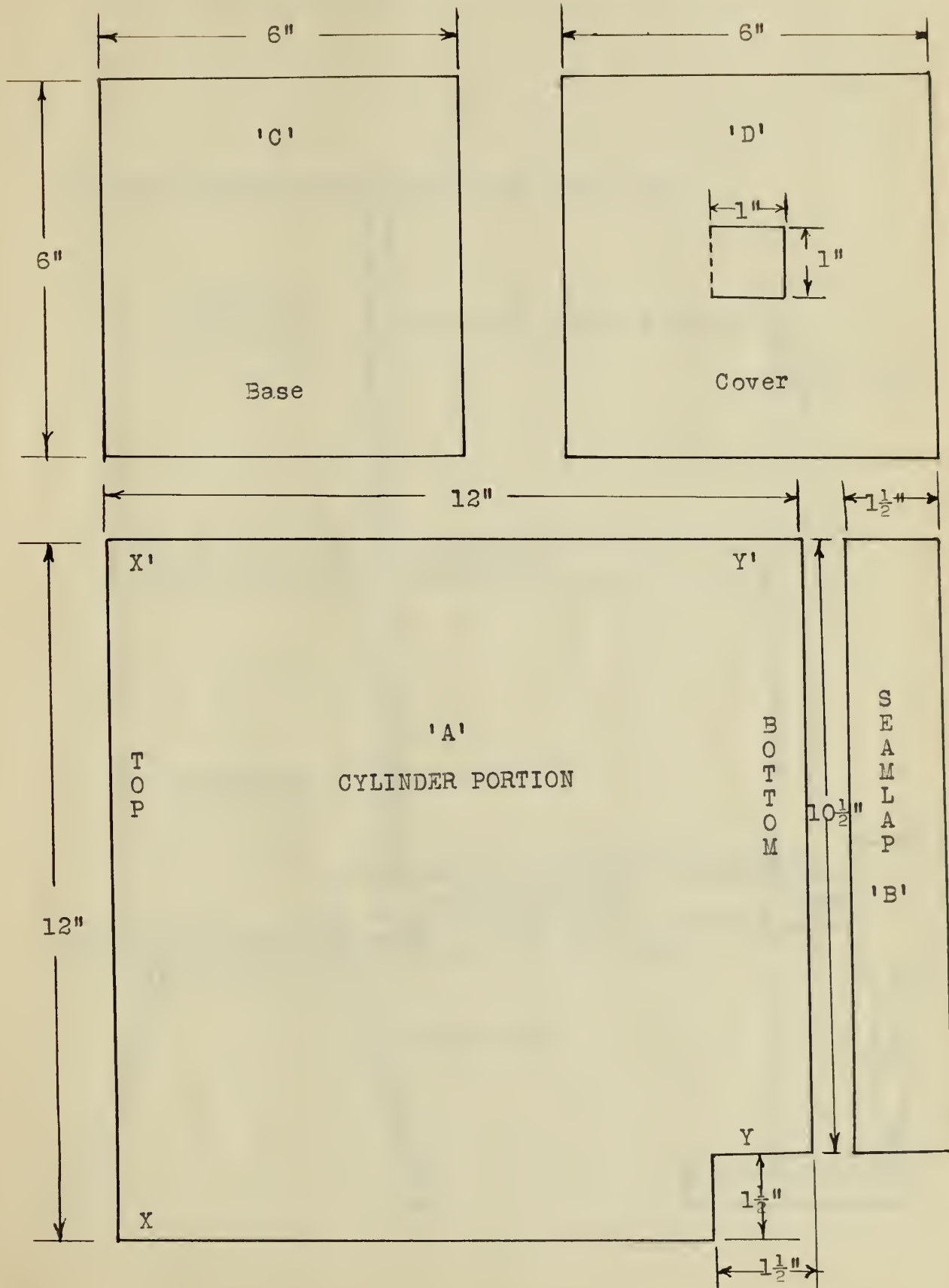
following experiments the size of the crucible is given.

Care should be exercised that the benches and the surrounding structures are protected against the heat radiated from the furnace when it is in operation. The tripod on which the furnace is set should in turn be set on a base of some material that is not easily affected by heat. The upright structures surrounding the furnace should be insulated against the heat by the erection of double walls of asbestos paper with a good sized air gap between each wall.

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DO hereby certify that the following is a true and correct copy of the original as the same appears in the records of the Department of the Interior.

TO ALL WHOM THESE PRESENTS SHALL COME, I GREETING.  
WHEREAS, the several Acts of Congress, in relation to the  
lands of the United States, have been amended and  
supplemented by subsequent Acts of Congress, and  
it is the policy of the Government to have a complete  
and correct copy of the same in the records of the  
Department of the Interior, and  
NOW KNOW YE, that the following is a true and correct  
copy of the original as the same appears in the records of the  
Department of the Interior.

ASBESTOS CYLINDER OVEN UNASSEMBLED





ASBESTOS CYLINDER FURNACE ASSEMBLED

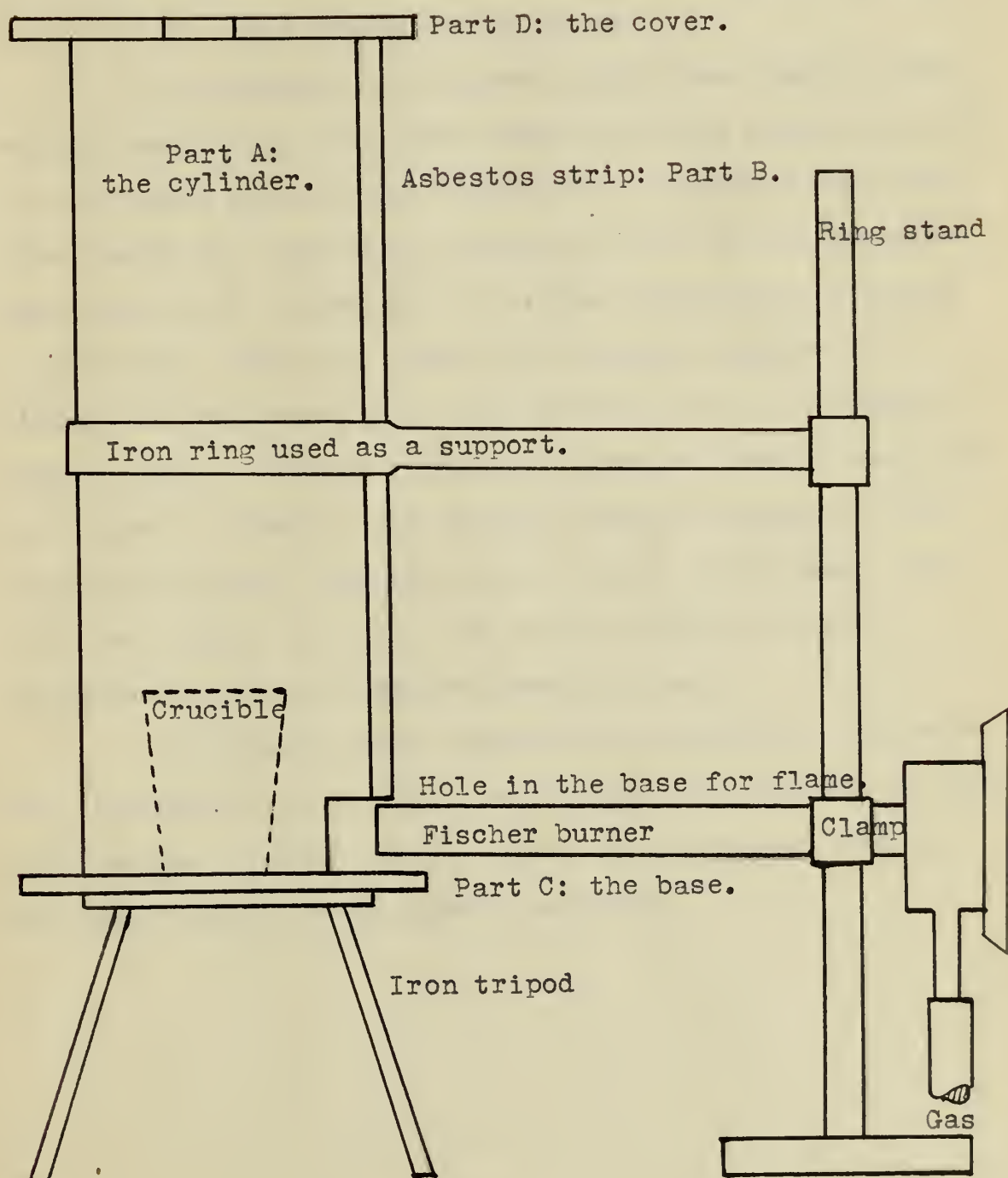
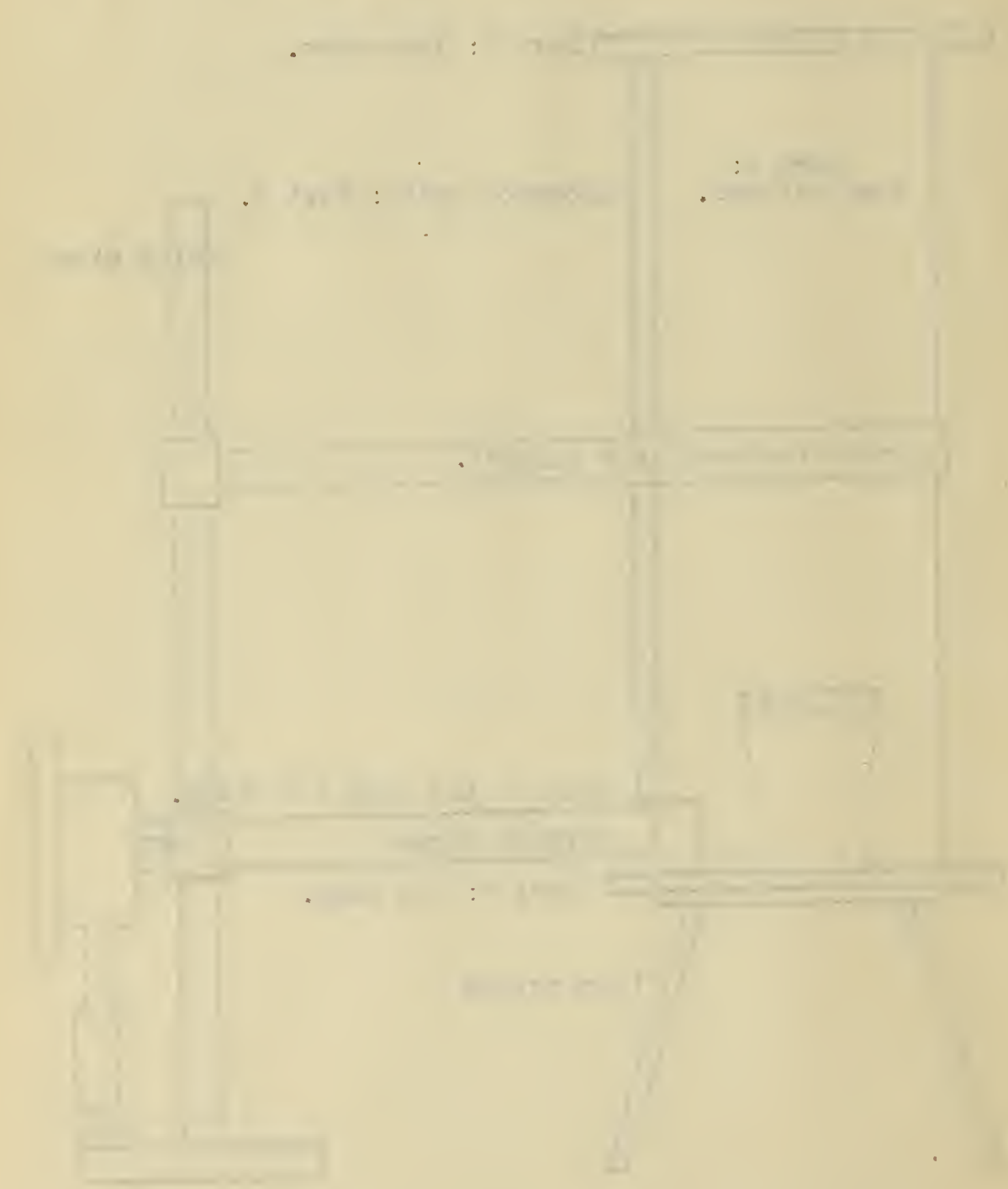


Diagram illustrating the structure of a building





GENERAL PROCEDURE FOLLOWED IN PREPARING COMPOUNDS  
IN THE NEW TYPE OF FURNACE

The following set of experiments was performed using the new type of gas furnace. Each of the experiments was performed several times in order to obtain the average yield and the best working conditions.

In general the following steps were used in preparing compounds in the new furnace: all the materials used in the fusion part of the experiment were ground to a very fine powder in a porcelain mortar and the various portions were then mixed intimately by further grinding in a porcelain mortar. When the powder had become a homogeneous mixture it was packed in a clay crucible until it formed a compact mass. (The butt end of a porcelain pestle was used as a tamp.) Finally, the crucible was set inside of the asbestos cylinder, exactly in the center of the base, the cover was placed over the top, and the Fischer burner adjusted to give the maximum heating effect.

In general this procedure is similar to the procedure followed in most fusion reactions. The details of these experiments, however, vary a great deal and most of the chemical work has been greatly modified.



PREPARATION OF COMPOUNDS IN THE NEW FURNACE

CALCIUM OXALATE FROM CALCIUM SULPHATE

Materials: 10.7 grams of plaster of Paris.  
5.0 grams of powdered wood charcoal.  
10.0 grams of oxalic acid.  
6.0N. hydrochloric acid.

Apparatus: Asbestos cylinder furnace.  
Hessian crucible three inches high and two inches wide.  
One 400 c.c. beaker.

Procedure: Unless the charcoal is already finely powdered grind it in a porcelain mortar. Add the plaster of Paris, which is also very finely powdered, to the charcoal in the mortar and mix the two materials intimately. Pack this mixture in a Hessian crucible and heat in an asbestos cylinder oven at a glowing heat for three hours. When cool inspect the contents of the crucible, particularly the inner portions where the heat would have penetrated the least; there should be no unburned carbon left; a small sample of the fused mass should dissolve with effervescence (hood) in hydrochloric acid and leave no residue. If the reduction has not been complete, grind the material again after adding five grams more of finely powdered wood charcoal and heat again for three hours. Repeat the above procedure until the

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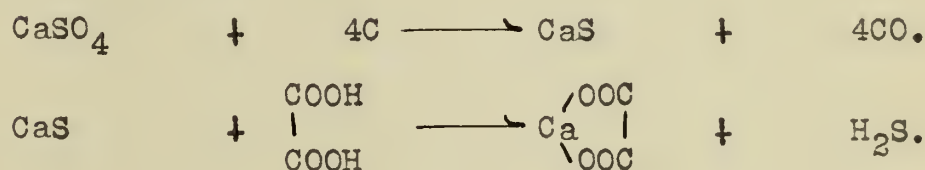
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hydrochloric acid procedure leaves no traces of residue other than a slight turbidity. In the meantime prepare a solution of ten grams of oxalic acid dissolved in fifty c.c. of water. When the heating or fusion process is completed, add the resulting mass, which has been powdered in a porcelain mortar, to the oxalic acid in a 400 c.c. beaker (hood). Care should be exercised at this point for the hydrogen sulphide is evolved quite rapidly. Allow the reaction to continue with occasional stirring until no more gas is evolved then boil the solution to drive off any hydrogen sulphide in solution. A small portion of the precipitate thus formed should not give a test for hydrogen sulphide when it is tested with hydrochloric acid. If a test is given then added oxalic acid solution should complete the reaction. This test is repeated until no more hydrogen sulphide results. Filter the solution after boiling and wash the residue three times with hot water. Dry the resulting product with paper towels.

Equations of reactions:







Percentage of yield:

Theoretical yield ..... 10.7 g.

Actual yield ..... 10.0 g.

Percentage yield ..... 93.0%.

In this experiment is shown the operating efficiency of the furnace in that the plaster of Paris was a chemically pure compound and therefore 100%  $\text{CaSO}_4$ . Some of the material was lost in the testing steps, however, that amount is quite small and would not interfere, largely, with the actual efficiency.



# CADMIUM SULPHIDE FROM CADMIUM SULPHATE

Materials: 10 grams of anhydrous cadmium sulphate.

5 grams of powdered wood charcoal.

30 c.c. of hydrochloric acid.

Ammonium hydroxide

Hydrogen sulphide generator.

Apparatus: Asbestos cylinder furnace.

Hessian crucible three inches high and two inches wide.

2 250 c.c. beakers.

Procedure: Be sure that the cadmium sulphide is anhydrous by heating in a porcelain dish over an open flame.

Unless the charcoal is already very finely powdered, grind it thoroughly in a mortar. Add the cadmium sulphate, finely powdered, to the wood charcoal in the mortar and mix intimately. Pack this mixture in a Hessian crucible and heat at a glowing heat for three hours in an asbestos cylinder oven. When cold note the color of the fused mass: a brick colored mass is noted here. Place this mixture in a mortar and powder it, then add this powder to a solution of hydrochloric acid made by adding fifteen c.c. of concentrated hydrochloric acid to fifteen c.c. of water. Boil this solution until no more hydrogen sulphide is generated and filter the resulting solution. The residue on the filter should contain no red or yellow materials.

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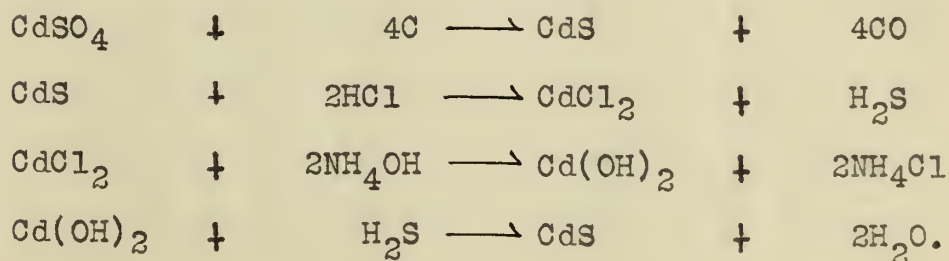
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If it does, boil the residue again with the same amount and strength of acid as previously and again filter, adding the two filtrates together and repeat the observation process until no yellow or red materials remain on the filter paper. Add all of the filtrates together. Make the filtrate neutral with ammonium hydroxide and then alkaline with the addition of ten c.c. more of ammonium hydroxide. Pass hydrogen sulphide through this solution until it becomes saturated. A yellow precipitate of cadmium sulphide should result. Filter and wash the precipitate with cold water. Test the filtrate for further cadmium by passing hydrogen sulphide into the filtrate, which, if not still alkaline should be made so by the addition of more ammonium hydroxide. If a precipitate results, add it to the first precipitate and repeat the hydrogen sulphide test with the second precipitate. Repeat this test until no further test is given for cadmium, washing each precipitate with cold water. The combined precipitates are dried with paper towels.

Equations of reactions:







Percentage of yield:

Theoretical yield ..... 6.0 g.

Actual yield ..... 6.0 g.

Percentage of yield ..... 87.0%.

The percentage in this experiment is lower than in the first due to the fact that a great many of the steps required the handling of the materials and a mechanical loss was unavoidable.

Very respectfully

Yours truly

Wm. L. G. ...

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# BARIUM NITRATE FROM BARYTE

Materials: 10 grams of powdered Baryte.

5 grams of wood charcoal.

6 N. Nitric acid.

Ethyl alcohol.

Apparatus: Hessian crucible three inches high and two inches wide.

Asbestos cylinder furnace.

Fischer burner.

Evaporating dish.

Procedure: Grind ten grams of finely powdered Baryte together with five grams of finely powdered wood charcoal until the mixture is intimate. Pack the mixture in a Hessian crucible and place on top of the mixture a layer of powdered wood charcoal. Heat the crucible in an asbestos cylinder furnace at a glowing heat for three hours. Cool the crucible, place the contents in a 400 c.c. beaker and add fifty c.c. of water. Stir the mixture and while stirring add twenty c.c. of 6 N. nitric acid a few c.c. at a time (hood). When the evolution of hydrogen sulphide has ceased add a few drops of nitric acid to some of the clear solution to ascertain whether or not the barium sulphide has been completely decomposed. If the evolution of gas continues, add a few more c.c. of nitric acid made up to the strength of fifty c.c.

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BY JOHN BURNET

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of water to twenty c.c. of 6 N. nitric acid. When the action has finally ceased, boil the solution to drive off any hydrogen sulphide in solution. Filter the resulting solution and evaporate the filtrate to about ten c.c. Permit the concentrated filtrate to cool. Crystallization should occur.

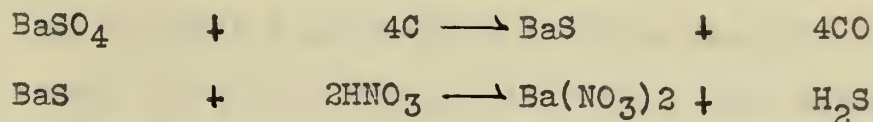
Clear octahedral crystals form. Collect these crystals and wash them with ethyl alcohol to remove impurities. A second crop of crystals may be obtained by concentrating the liquor from the first crystals by evaporation and then permitting cooling. The crystals that form are collected and redissolved in twenty c.c. of water and this solution is evaporated to about six c.c. The solution is allowed to cool and the crystals so formed are collected, dried by means of paper towels, and then added to the first crystals collected.

The above procedure is necessary because of the nature of the materials in the mineral Barytes. A qualitative analysis of this mineral for metallic ions revealed the presence of barium, strontium, and calcium. The qualitative analysis of the first crop of crystals revealed the presence of barium only. However, the analysis of the first crystallization of the second crop of crystals



revealed barium and strontium. It was necessary to remove the strontium by further fractional crystallization. The first fraction of the second crystallization had only barium present. This crop of crystals was, therefore, added to the first crop of crystals.

Equations of reactions:



Percentage yield from Barytes:

Theoretical yield from $\text{BaSO}_4$ .....	11.4 g.
Actual yield from Barytes .....	7.6 g.
Percentage yield .....	66.7%

As a further check on the efficiency of the furnace a quantitative analysis of the Barytes was made by the following method: a sample of finely powdered Barytes was weighed accurately on an analytical balance and then mixed with five times its weight of finely powdered anhydrous sodium carbonate. The mixture was placed in a platinum crucible and fused until a clear melt formed. The crucible was allowed to cool and the resulting product taken out and pulverized to a fine powder. Care was taken that the entire melt was removed without leaving a trace of the melt in the crucible. The powder was placed in a flask containing two hundred and fifty c.c. of hot





water. The solution was stirred and then filtered by means of suction. The residue was washed on the filter paper with hot water until the washings gave no test for sulphates when added to a barium chloride solution. The residue was then dissolved in enough six normal nitric acid to place all of the material in solution and the filtrate was received in a one hundred and twenty five c.c. Erlenmeyer flask. The procedure from here on was taken from Treadwell and Hall, "Analytical Chemistry", Volume Two, Quantitative, Fifth Edition, John Wiley and Sons, Inc., New York, New York, 1919, pages 79-81.

The following results were obtained:

Weight of Barytes samples.....	0.473g.	0.497g.
Weight of CaO.....	0.002g.	0.002g.
Weight of BaCrO <sub>4</sub> .....	0.454g.	0.477g.
Weight of SrSO <sub>4</sub> .....	0.047g.	0.048g.
Percent of CaSO <sub>4</sub> in Barytes....	1.05%	1.0%
Percent of BaSO <sub>4</sub> in Barytes....	88.00%	88.00%
Percent of SrSO <sub>4</sub> in Barytes....	<u>9.85%</u>	<u>9.70%</u>
Total percentage.....	98.90%	98.70%

The remainder of the percentage is divided between iron and aluminum oxides.





The efficiency of the furnace calculated from these figures is as follows:

Actual weight of $\text{BaSO}_4$ .....	8.8 g.
Theoretical yield from $\text{BaSO}_4$ .....	9.85 g.
Efficiency of the furnace .....	89.5%

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DIVISION OF THE PHYSICAL SCIENCES  
DEPARTMENT OF CHEMISTRY

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BARIUM HYDROXIDE FROM BARYTE

Materials: 10 grams of powdered Baryte.

5 grams of wood charcoal.

8 grams of copper oxide.

Apparatus: Asbestos cylinder furnace.

Hessian crucible three inches high and two inches wide.

Three inch evaporating dish.

Procedure: Mix ten grams of finely powdered Baryte thoroughly with five grams of finely powdered wood charcoal. Pack the mixture in a Hessian crucible and heat at a glowing heat for three hours in an asbestos cylinder furnace. Cool the fused mass and place it in a 400 c.c. beaker. Add to the beaker sixty c.c. of water and boil the solution. Place the resulting solution in an evaporating dish and boil. While the solution is hot add to it eight grams of powdered copper oxide. Permit the precipitate to settle. So long as a yellow color persists barium sulphide still exists in solution. This color may be observed by noticing the color of the solution against the white background of the porcelain evaporating dish. If the yellow color still persists add more copper oxide and boil. As soon as the yellow color disappears filter the hot solution as rapidly as possible. Crystals of barium hydroxide separate very rapidly

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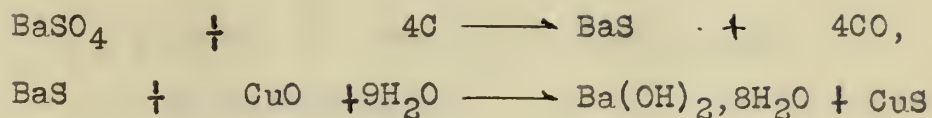


from the cool solution and clog the pores of the filter. In order to prevent this add enough water to the evaporating dish to replace the water lost in evaporation and pour the hot solution through an ordinary filter by allowing the liquid to run from the evaporating dish down a stirring rod that is held perpendicular to the bench and in contact with the lip of the evaporating dish and thence into the opening of the filter funnel keeping the solution in the beaker hot while waiting for the portion in the funnel to filter. Catch the filtrate in a 100 c.c. flask and allow the residue to remain in the evaporating dish. Add ten c.c. of water to the residue and stir thoroughly. Heat for a moment over a flame and then pour the residue and solution into the filter funnel and drain off all of the liquid. Stopper the retaining flask to shut off the air and wrap the whole, stopper and flask, in a towel, so it may cool slowly and larger crystals form. Finally, after several hours, cool the solution by running cold water from a tap over the flask. Collect the crystals on a suction filter taking care not to suck too much air through the crystals as they react very easily with carbon dioxide in the air. Wrap the product in a paper towel and allow to dry over-



night at room temperature.

Equations of reactions:



Percentage of yield from Baryte:

Theoretical yield from BaSO <sub>4</sub> .....	13.6 g.
Actual yield from Baryte .....	7.5 g.
Percentage yield .....	55.9%

In this experiment the efficiency of the yield is again, apparently, low. As is the case in the preceding experiment, the ore used is not 100% BaSO<sub>4</sub>. The closeness of the low percentage yield tells the whole story; the ore used in these two experiments were from the same source, and it is, therefore, only natural the the efficiencies of these two experiments should be affected similarly.

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### STRONTIUM NITRATE FROM CELESTITE

Materials:        10 grams of powdered Celestite.  
                    5 grams of powdered wood charcoal.  
                    6 N. Nitric acid.  
                    Ethyl alcohol.

Apparatus:        Asbestos cylinder furnace.  
                    Hessian crucible three inches high and two  
                    inch wide.  
                    Three inch evaporating dish.

Procedure:        Mix ten grams of finely powdered Celestite  
                    intimately with five grams of finely powdered  
                    wood charcoal. Pack the mixture in a Hessian  
                    crucible and heat at a glowing heat for three  
                    hours in an asbestos cylinder furnace. Cool the  
                    crucible and place the contents in a four hundred  
                    c.c. beaker. Add fifty c.c. of water, stir, and  
                    add twenty c.c. of 6 N. nitric acid a few c.c. at  
                    a time with constant stirring, (hood). Permit  
                    the reaction to continue until no more hydrogen  
                    sulphide gas is given off. Add a few more c.c.  
                    of nitric acid to the solution to ascertain whether  
                    or not the decomposition of the strontium sulphide  
                    is complete. If the action continues, add a few  
                    more c.c. of nitric acid made to the concentration  
                    of fifty c.c. of water to twenty c.c. of 6 N. nitric  
                    acid. When the action finally ceases, boil the  
                    solution in order to drive off any hydrogen sulphide



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RESEARCH REPORT

NO. 100

1955

BY

DR. J. H. GOLDSTEIN

AND

DR. R. M. MAYER

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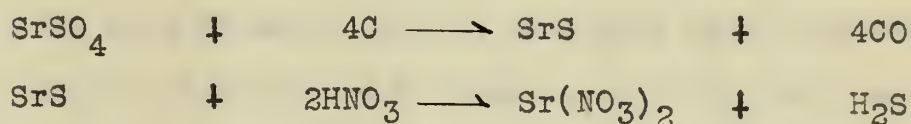
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in solution. Filter the resulting solution while still hot and evaporate the filtrate to ten c.c. over an open flame and then permit the concentrated filtrate to cool. Crystallization should occur. Wash these crystals with ethyl alcohol to remove any impurities.

A further crop of crystals may be collected by further concentration of the filtrate from above.

Equations of reactions:



Percentage of yield from Celestite:

Theoretical yield from $\text{SrSO}_4$ .....	11.5 g.
Actual yield from Celestite .....	4.6 g.
Percentage yield .....	40.0%

Recrystallization was not necessary for the second crop of crystals in this experiment because a qualitative analysis of the mineral revealed strontium in large quantities and calcium only as a trace. The analysis of the second crop of crystals showed only strontium present.

Figure 1. The effect of the concentration of the *Agaricus bisporus* spores on the growth of *Agaricus bisporus* on the substrate.

Figure 1. The effect of the number of trials on the number of correct responses. The number of correct responses was significantly higher than the number of incorrect responses for all groups. The number of correct responses was significantly higher than the number of incorrect responses for all groups. The number of correct responses was significantly higher than the number of incorrect responses for all groups.

Figure 1. The 100 most cited articles in the field of the study of the effects of the environment on the development of children. The figure shows the first 100 articles in the field of the study of the effects of the environment on the development of children, ranked by citation count. The articles are listed in descending order of citation count, with the most cited article at the top. The citation count for each article is shown in the rightmost column. The articles are grouped into three categories: 'Developmental Psychology', 'Pediatrics', and 'Psychiatry'. The 'Developmental Psychology' category includes 40 articles, 'Pediatrics' includes 30 articles, and 'Psychiatry' includes 30 articles. The articles are listed in descending order of citation count, with the most cited article at the top. The citation count for each article is shown in the rightmost column. The articles are grouped into three categories: 'Developmental Psychology', 'Pediatrics', and 'Psychiatry'. The 'Developmental Psychology' category includes 40 articles, 'Pediatrics' includes 30 articles, and 'Psychiatry' includes 30 articles.

# STRONTIUM HYDROXIDE FROM CELESTITE

Materials: 10 grams of powdered Celestite.  
5 grams of powdered wood charcoal.  
8 grams of powdered copper oxide.

Apparatus: Asbestos cylinder furnace.  
Hessian crucible three inches high and two inches wide.  
Porcelain evaporating dish.  
400 c.c. beaker.

Procedure: Mix ten grams of powdered Celestite intimately with five grams of finely powdered wood charcoal. Pack the mixture in a Hessian crucible and heat at a glowing heat for three hours in an asbestos cylinder furnace. Cool and place the contents of the crucible in a 400 c.c. beaker. Add sixty c.c. of water. Heat the solution to boiling and filter while hot. A yellowish solution results. This is placed in a porcelain dish, heat to boiling, and then add eight of powdered copper oxide. Permit the precipitate to settle. So long as any strontium sulphide is present a yellow color will persist, which may be observed by noticing the color of the solution against the white background of the porcelain dish. If the color persists boil the solution until it disappears. Crystals of strontium hydroxide separate very rapidly from this solution as it cools, hence it must be filtered rapidly in order to prevent

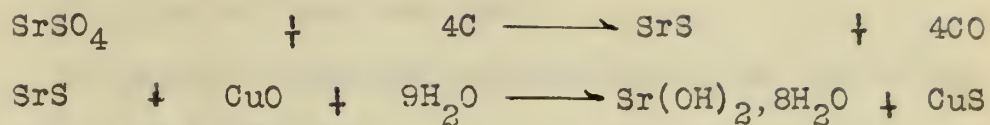




them from clogging the pores of the filter paper. Add enough water to the evaporating dish to make up for any water lost in evaporation, heat the solution to boiling, and pour, while hot, into an ordinary filter by allowing the liquid to run down a stirring rod held perpendicular to the bench and in contact with the lip of the evaporating dish and thence to the filter funnel, keeping the solution in the beaker hot while waiting for the portion in the funnel to filter. Catch the filtrate in a 100 c.c. flask and allow the residue to remain in the evaporating dish. Add ten c.c. of hot water into the evaporating dish, stir, and pour the entire mass into the filter funnel. Stopper the flask containing the combined filtrates from the foregoing procedure and wrap the whole, stopper and flask, in a towel and permit slow cooling in order that large crystals may form. Finally, after several hours, place the flask under a flow of cold water from a tap and then collect the resulting crystals by suction filtration, taking care not to suck too much air through the crystals as they react very readily with the carbon dioxide in the air. Wrap the product in paper towels and allow to dry at room temperature overnight.



Equations of reactions:



Percentage of yield from Celestite:

Theoretical yield from $\text{SrSO}_4$ .....	6.65 g.
Actual yield from Celestite .....	3.0 g.
Percentage of yield .....	45.0%

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### BERYLLIUM OXIDE FROM BERYL

- Materials:** 10 grams of powdered Beryl.  
10 grams of powdered anhydrous sodium carbonate.  
Sodium bicarbonate.  
Dilute hydrochloric acid.
- Apparatus:** Asbestos cylinder furnace.  
Clay crucible four inches high and two and a half inches wide.  
Asbestos pad.  
Porcelain mortar and pestle.  
Suction flask and filter funnel.  
2 400 c.c. beakers.
- Procedure:** Grind the finely powdered Beryl and the finely powdered sodium carbonate, which must be anhydrous, together in a mortar with a pestle until they form an intimate mixture. Pack the mixture in a clay crucible, and heat at a glowing heat in an asbestos cylinder furnace for three hours. At the end of that time pour the red hot melt on to an asbestos pad and allow to cool in the open air. When the melt has cooled break it into small pieces and grind these pieces to a fine powder in a porcelain mortar with a porcelain pestle. Extract the soluble materials by washing the powder in a beaker with fifty c.c. of water. Boil the solution and stir while boiling. Filter off the hot liquid by means of a suction flask and funnel. Repeat this washing process twice again



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OF THE UNIVERSITY OF OXFORD

IN TWO VOLUMES

VOLUME THE FIRST

1680

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using the residue in each step as the powder in the first step. While the powder is on the filter paper after the third washing pour through it twenty-five c.c. of hot water and then suck dry on the filter paper. Dissolve this residue in sufficient six normal hydrochloric acid to make an acid solution of the metallic ions present in the powder. At this point all of the powder should be in solution. If this is not true then filter the solution using a hardened filter paper and suction filtration. The clear solution should be tested for phosphates and if found present should be removed by the regular qualitative procedure. The solution should be just barely acid and is added to a solution of sodium bicarbonate of such a strength that when the acid solution is added the combined solutions will be about ten percent sodium bicarbonate. The combined solutions should be contained in a tall beaker which is covered with a watch glass immediately after the addition of the acid solution. The solution is brought to a quick boil (a thermometer should be used to determine the boiling as the bubbles of carbon dioxide may be mistaken for boiling) and allowed to boil for three minutes. The hot solution is cooled as quickly as possible by allowing cold tap water to run around the base of the beaker. Wash the residue three times with hot water after filtering and combine

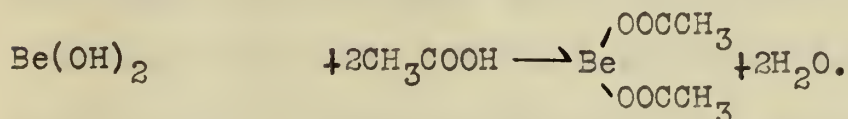
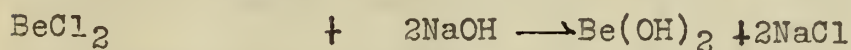
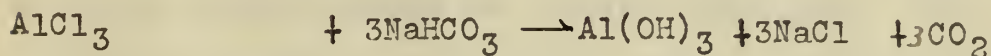
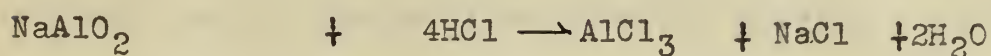
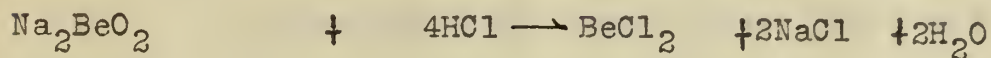
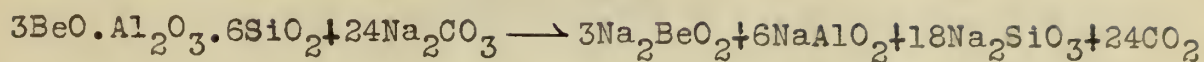


the resulting filtrates. Treat the residue obtained here again as the first residue was treated with the sodium bicarbonate solution and combine the filtrates obtained here with the first filtrates obtained from the sodium bicarbonate treatment. Neutralize this solution with sodium hydroxide solution and then make it alkaline with the addition of an excess of sodium hydroxide. Filter the solution thus formed and dry the residue with paper towels. This precipitate is almost entirely beryllium oxide. To remove the last traces of impurities redissolve this powder in six normal hydrochloric acid and repeat the entire foregoing process from the addition of the sodium bicarbonate solution. To the final residue add enough dry acetic acid to dissolve the entire product. Evaporate to dryness in a porcelain dish (hood). Dissolve this residue in chloroform, place in a separatory funnel, add an equal amount of water, and shake the entire mass vigorously. Drain off the chloroform layer and repeat the washing process in the separatory funnel. After the second washing evaporate the chloroform solution to dryness on a water bath using a condenser to collect the chloroform for further use. The final product is beryllium acetate.

The first thing I noticed when I stepped out of the car was the cold. It was a sharp contrast to the warm blanket I had been sitting under. I looked up at the sky, which was a pale, hazy blue. The air was still, and the only sound I could hear was the distant hum of traffic. I took a deep breath, feeling the cold air fill my lungs. I was alone in the world, and it felt like I had been thrown into a new one. I walked slowly, my feet sinking into the soft ground. The sun was low in the sky, casting a long, golden glow over the landscape. I felt a sense of peace, a sense of being in the right place at the right time. I closed my eyes and let the world wash over me. I was home.



Equations of reactions:



Percentage of yield from Beryl:

Theoretical yield from Beryllium Oxide.... 1.4 g.

Actual yield of Beryllium Oxide figured

from the Beryllium Hydroxide..... 1.0 g.

Percentage of yield ..... 71.5%

Theoretical yield of Beryllium Acetate

from Beryllium Hydroxide ..... 5.0 g.

Actual yield ..... 4.0 g.

Efficiency ..... 80.0%

TABLE 1. - SUMMARY OF DATA

Year	Month	Day	Time	Location	Remarks
1954	June	1	10:00	1000	1000
1954	June	1	11:00	1000	1000
1954	June	1	12:00	1000	1000
1954	June	1	13:00	1000	1000
1954	June	1	14:00	1000	1000
1954	June	1	15:00	1000	1000
1954	June	1	16:00	1000	1000
1954	June	1	17:00	1000	1000
1954	June	1	18:00	1000	1000
1954	June	1	19:00	1000	1000
1954	June	1	20:00	1000	1000
1954	June	1	21:00	1000	1000
1954	June	1	22:00	1000	1000
1954	June	1	23:00	1000	1000

TABLE 2. - SUMMARY OF DATA

1954	June	1	10:00	1000	1000
1954	June	1	11:00	1000	1000
1954	June	1	12:00	1000	1000
1954	June	1	13:00	1000	1000
1954	June	1	14:00	1000	1000
1954	June	1	15:00	1000	1000
1954	June	1	16:00	1000	1000
1954	June	1	17:00	1000	1000
1954	June	1	18:00	1000	1000
1954	June	1	19:00	1000	1000
1954	June	1	20:00	1000	1000
1954	June	1	21:00	1000	1000
1954	June	1	22:00	1000	1000
1954	June	1	23:00	1000	1000

## CONCLUSIONS

In concluding and summarizing, the new type of furnace may be very well adapted for usage in the place of blast lamp furnaces for fusion reactions. The efficiency of the furnace is high. This is shown by the percentage yield results of the experiments in which pure compounds were used; see the percentage yield of calcium oxalate from calcium sulphate and the percentage yield of cadmium sulphide from cadmium sulphate on pages 15 and 18 respectively.

The lasting or wearing properties of the furnace is not very notable. Of course the durability of the furnace depends on how well it is constructed. In general, the furnaces used in these experiments had to be replaced after about thirty hours of constant heating usage. Of course this is not a handicap to the economical usage of the furnace in as much as the materials used are very low in price.

CHAPTER IV

The first of the two principal questions which arise in connection with the study of the history of the English language is the question of the origin of the language. The second question is the question of the development of the language. The third question is the question of the influence of the language on the culture of the English people. The fourth question is the question of the influence of the culture of the English people on the language. The fifth question is the question of the influence of the language on the literature of the English people. The sixth question is the question of the influence of the literature of the English people on the language. The seventh question is the question of the influence of the language on the science of the English people. The eighth question is the question of the influence of the science of the English people on the language. The ninth question is the question of the influence of the language on the art of the English people. The tenth question is the question of the influence of the art of the English people on the language.

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